Corrosion protective layer for substrate against high temp. carburising atmos

molten carbonate

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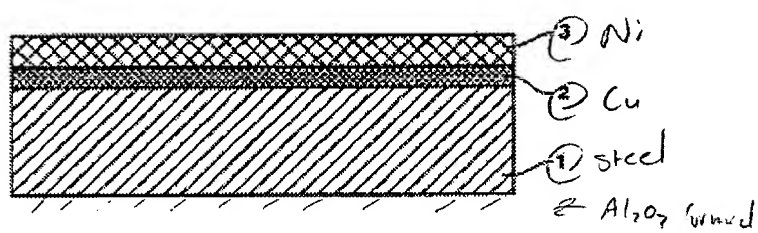
Abstract of **DE19523637**

A substrate (1) is provided with a corrosion protective coating by initially coating with a non-diffusion layer (2) of thickness 0.2-6, pref. 0.5-3 mu m followed by a corrosion resistant layer (3) of thickness 0.2-10, pref. 0.5-5 mu m.



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The available invention concerns generally an anti-corrosive coating for applications in more carburizing (more reducing) atmosphere at high temperatures and a procedure for the production of such, as well as an energized construction unit for a fusion carbonate gas cell, provided with an anti-corrosive coating, like in particular an anode current collector or a bipolar plate of a fusion carbonate gas cell. In the anode region to a fusion carbonate gas cell conditions prevail - carburizing atmosphere and low oxygen partial pressure as well as presence of lithium and. Potassium carbonate melts - which to a rapid corrosion of high-grade steel components planned in the fusion carbonate gas cells lead. This corrosion is substantially accelerated by the high temperatures dominant with the enterprise of fusion carbonate gas cells. The reason for this is to be seen in the fact that in the carburizing atmosphere formed the oxide coatings, contrary to such, are which are formed in an oxidizing atmosphere not close and stable and therefore the used highly alloyed high-grade steel to protect ability those does not forbid itself often selected use of aluminum-bearing steel or the Aluminieren of the steel for in fusion carbonate gas cells used the energized parts, thus in particular to anode current collectors or bipolar plates, because of the very high electrical resistance of the developing oxide coatings. A further problem insists in creeping the melted salts electrolytes on such metallic construction units. This creeping is one of the loss mechanisms electrolytes and works therefore life span-limiting. Besides creeping favours the contamination of a fission gas reaction catalyst planned for the enterprise of the fusion carbonate gas cells and not possibly makes thereby the employment of a direct internal reformation, which would be energetically particularly favourable. So far the corrosion of the high-grade steel components like also a creeping of the melted salts on the metallic construction units in the anode region by fusion carbonate gas cells by a coating of the high-grade steel sheet metals by nickel were prevented. Nickel is inert in the atmosphere contained in the anode region and by the melt is not moistened. Coating the construction units with nickel is done with flat components e.g. with plating and with construction units with a three-dimensional surface via galvanic coating. However so far not solved and/or solvable difficulties do not arise also here. Like that are wide nickel-plated sheet metals with width of more than 0.5 m world-wide hardly available, there the roll-bonded cladding of such broad volumes high requirements to the rolling mill of place-galvanically nickel plated parts generally show the problem that the formed layer exhibits pores. In order to prevent pores, galvanically applied nickel in the thinnest place must be thick at least 50 mu m. With construction units with a three-dimensional form, as a anode-laterally used current collector can at best a dickenverhaeltnis of 2 1 from the thickest to the thinnest place are reached. In order to exclude with such dickenverhaeltnis places with a smaller thickness than 50 mu m, the nickel need for a square meter of current collector surface amounts to about 2.5 kilograms. This means a need of 1.000 tons nickel for a production desired large quantities of nickel, necessary by fusion carbonate gas cells of 400 MW/a.Solche, impairs the economy of fusion carbonate gas cell production, in particular when rising the strongly varying nickel prices. This applies in particular to galvanic nickel plating of the current collectors, for which sulfur-poor Sulfamatnickel is necessary. Further it is difficult, with larger surfaces, like with at present with a half square meter lying the cell area, when applying thick galvanic nickels laminates close tolerance to keep.

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Die vorliegende Erfindung betrifft allgemein eine Korrosionsschutzbeschichtung für Anwendungen in aufkohlender (reduzierender) Atmosphäre bei hohen Temperaturen und ein Verfahren zur Herstellung einer solchen, sowie ein mit einer Korrosionsschutzbeschichtung

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Finally galvanic manufacturing of nickel layers is increasingly problematic and/or uneconomic in the large yardstick by the associated environmental impact and itself the official editions resulting in from it. In principle the thinnest attainable closed layer for the corrosion protection and as creep barrier is sufficient, if it is inert under the given conditions and further the sufficient electrical conductivity necessary for the gas cell enterprise has. With the operatings temperature of fusion carbonate gas cells from 600 to 700 DEG C the diffusion rate is however so high that nickel layers of at least 50 mu m are necessary thickness, in order to achieve the life span desired from 40.000 hours to. The task of the invention is thus, an anti-corrosive coating for applications in more carburizing it (more reducing) atmosphere at high temperatures, in particular an energized construction unit as to be indicated an anode current collector or a bipolar plate of a fusion carbonate gas cell and a procedure for the production of it, with which with a small quantity of used up coating material a sufficiently high life span is reached. This task is solved according to invention by the fact that on the substrate which can be coated a thin layer of a not diffusing material is formed as diffusion barrier layer, and that on the diffusion barrier layer a thin layer of a material corrosion resistant is formed as corrosion protection layer. In accordance with an alternative solution of the task posed a only one thin layer from material corrosion resistant and a not rusting can be formed as corrosion protection and diffusion barrier layer on the substrate. Favourable training further of the invention are indicated in the unteranspruechen. In the following remark examples of the invention are described on the basis the design. The figure shows schematizes a cross section opinion of a substrate provided with an anti-corrosive coating in accordance with the invention. In the figure the reference symbol 1 means a substrate, on which an anti-corrosive coating is applied, which consists of a diffusion barrier layer 2 and a corrosion protection layer 3, whereby the corrosion protection layer 3 can work at the same time also as creep barrier. The substrate 1 can be generally a construction unit, which is exposed to a reducing, carburizing atmosphere at high temperatures. In particular the substrate 1 can be a construction unit used consisting of high-grade steel in the anode region of fusion carbonate gas cells, thus in particular the anode current collector or the bipolar plate. For the coating according to invention is applicable in particular the anode side of the bipolar plate. In accordance with a first remark example of the invention a diffusion barrier layer 2 from TiN is applied on a substrate 1 made of high-grade steel. Over the diffusion barrier layer 2 a nickel layer is applied as corrosion protection layer 3. The diffusion barrier layer 2 and the corrosion protection layer 3 have in each case a thickness of few micrometers, with this remark example have the diffusion barrier layer 2 a thickness of 0,5 to 3 mu m and the corrosion protection layer 3 a thickness from 0,5 to 5 mu m. both the diffusion barrier layer 2 and the corrosion protection layer 3 are applied by ferries of the thin-film technology on the substrate 1. As ferries knows sputtering, a Arc procedure, vaporizing, ion Plating, CVD or a similar procedure uses both layers can by the same procedure or by different procedures on the substrate 1 be separated. A particularly good adhesion of the layers is reached, if these are formed at increased substrate temperatures, approximately with 400 DEG C to 700 DEG C. However also during a schichtbildung without additional heating a satisfying adhesion of the layers on the substrate can be already reached. For the adjustment of the plasma and/or the material stream to the construction unit in the sense of an even layer thickness, which can be coated flowing in the plasma, appropriate magnetic and/or electrical fields can be produced during the schichtbildung by means of additional permanent or coil magnets intended in the coating device and/or additional electrodes.

nickel is oxidation-resistant?

.5-3 um (layer 2)
.5-5 um (layer 3)

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Schliesslich ist das galvanische Herstellen von Nickelschichten im grossen Massstab durch die damit verbundene Umweltbelastung und die sich daraus ergebenden behördlichen Auflagen zunehmend problematisch bzw. unwirtschaftlich.

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In accordance with further remark examples of the invention also another material can be used, which is inert in the regarded atmosphere, like outer ones, cu, Pt, Ru or Pd. alternative for TiN can than material for the diffusion barrier layer 2 also CrN, ZrN, TiCN or like be used for the corrosion protection layer 3 in place of nickel. These layers can be formed likewise through one of the procedures of the thin-film technology indicated above. In accordance with remark variation in type of the invention a only one thin layer from at the same time material corrosion resistant and a rustproof than corrosion protection and diffusion barrier layer with a thickness can be preferably trained from 0,2 to 10 mu m, from 0,5 to 5 mu m from TiN, CrN, TiNi or the like on the substrate. Also this layer is formed by a procedure of the thin-film technology. Both the diffusion barrier layer 2 intended in connection with a separate corrosion protection layer 3 and the only thin corrosion protection and diffusion barrier layer can be formed in accordance with remark variation in type also by chromoxid. This chrome oxide coating can be produced for out chromhaltigem high-grade steel by glowing of an existing substrate in an oxygen-containing atmosphere with 800 to 1,000 DEG C. That oxygen content is adjusted thereby to a suitable value. Because of the high oxygen affinity of the chrome primarily close chromoxid with the glowing, which a outstanding barrier layer form in accordance with a further remark example of the invention with an anti-corrosive coating provided an energized construction unit for a fusion carbonate gas cell, like an anode current collector or a bipolar plate production, with on a substrate 1 a diffusion barrier layer 2 from TiN with a thickness, consisting of high-grade steel, from 0,2 to 10 mu m, preferably 0.5 to 3 mu m is trained, develops. On the diffusion barrier layer 2 a corrosion protection layer 3 made of nickel with a thickness is from 0,2 to 10 mu m, preferably 0.5 to 5 mu m applied. The layers are formed by a procedure of the thin-film technology, like a sputtering, a Arc procedure, a vaporizing, ion Plating, CVD or a similar procedure. In order to achieve a close, well responsible layer, applying at high substrate temperatures, about 400 DEG C to 700 DEG C accomplish however also during a schichtbildung without additional heating can preferential a satisfying adhesion of the layers on the substrate be already reached. For the adjustment of the plasma and/or the material stream to the construction unit in the sense of an even layer thickness, which can be coated flowing in the plasma, magnetic and/or electrical fields can be produced during the schichtbildung by means of additional, in the coating device of intended permanent or coil magnets and/or additional electrode-appropriate. For the diffusion barrier layer 2 can be used alternatively for TiN also CrN, ZrN, TiNi or TiCN. Further the diffusion barrier layer can be formed 2 also by chromoxid, which is accomplished by glowing of the chromhaltigen high-grade steel sheet metal in an oxygen-containing atmosphere with 800 to 1,000 DEG C. In place of nickel can be used for the corrosion protection layer also outer one, cu, Pt, Ru or Pd. -- ---------- DATA supplied from the DATA cousin esp@cenet - Worldwide

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Gemäss weiteren Ausführungsbeispielen der Erfindung kann für die Korrosionsschutzschicht 3 anstelle Nickel auch ein anderes Material verwendet werden, das in der betrachteten Atmosphäre inert ist, wie Au, Cu, Pt, Ru oder Pd. Alternativ zu TiN kann als Material für

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In English:

1. Procedures for the production of an anti-corrosive coating for applications in more carburizing (more reducing) atmosphere at high temperatures, with which the anti-corrosive coating on a substrate is trained, by it marked that on the substrate (1) a thin layer of a not-diffusing material is formed as diffusion barrier layer (2), and that on the diffusion barrier layer (2) a thin layer of a material corrosion resistant as corrosion protection layer (3) it is formed. 2. Procedure according to requirement 1, by the fact characterized that the corrosion protection layer (3) in a thickness is formed from 0,2 mu m to 10 mu m. 3. Procedure according to requirement 1, by the fact characterized that the corrosion protection layer (3) in a thickness is formed from 0,5 mu m to 5 mu m. 4.Verfahren according to requirement 1, 2 or 3, by the fact characterized that the diffusion barrier layer (2) in a thickness is formed from 0,2 mu m to 6 mu m. 5. Procedure according to requirement 1, 2 or 3, by the fact characterized that the diffusion barrier layer (2) in a thickness is formed for m from 0,5 mu m to 3 mu. 6. Procedure after the generic term of the requirement 1, by the fact characterized that on the substrate (1) a only one thin layer from material corrosion resistant and a rustproof is formed as corrosion protection and diffusion barrier layer (3) in a thickness from 0,2 mu m to 10 mu m, preferably 0.5 mu m to 5 mu m. 7. Procedure after one of the requirements 1 to 6, by the fact characterized that the corrosion protection layer (3) and/or the diffusion barrier layer (2) are formed by a thin section procedure. 8.Verfahren according to requirement 7, by the fact characterized that the corrosion protection layer (3) and/or the diffusion barrier layer (2) are formed by sputtering, a Arc procedure (Arc evaporation, Arc Bonding Sputtering), vaporizing, ion Plating or a CVD procedure. 9. Procedure according to requirement 7 or 8, by the fact characterized that the schichtbildung takes place at increased substrate temperature. 10. Procedure according to requirement 9, by the fact characterized that the schichtbildung takes place with 200 DEG C to 900 DEG C. 11 Procedure according to requirement 9, by the fact characterized that the schichtbildung takes place with 400 DEG C to 700 DEG C. 12. Procedure according to requirement 7 or 8, by the fact characterized that the schichtbildung without additional heating takes place. 13.Verfahren after one of the requirements 7 to 12, by the fact characterized that during the schichtbildung by means of additional permanent or coil magnets and/or additional electrodes magnetic and/or electrical fields are produced for the adjustment of the plasma to the construction unit which can be coated. 14. Procedure after one of the requirements 1 to 13, by the fact characterized that the substrate (1) consists of a highly alloyed rustproof steel. 15. Procedure after one of the requirements 1 to 14, by the fact characterized that the corrosion protection layer (3) made of nickel is formed. 16. Procedure after one of the requirements 1 to 14, by the fact characterized that the corrosion protection layer (3) from outer ones, cu, Pt, Ru or Pd is formed. 17.Verfahren after one of the requirements 6 to 14, by the fact characterized that the only corrosion protection and diffusion barrier layer (3) from TiN or CrN are formed. 18. Procedure after one of the requirements 1 to 5 or 7 to 1 7, by the fact characterized that the diffusion barrier layer (2) from TiN is formed. 19. Procedure after one of the requirements 1 to 5 or 7 to 17, by the fact characterized that the diffusion barrier layer (2) from CrN, ZrN, TiNi or TiCN is formed. 20 Procedure after one of the requirements 1 to 17, by the fact characterized that the diffusion barrier layer (2) or the only corrosion protection and diffusion barrier layer (3) made of chromoxid is formed.

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1. Verfahren zur Herstellung einer Korrosionsschutzbeschichtung für Anwendungen in aufkohlender (reduzierender) Atmosphäre bei hohen Temperaturen, bei dem die Korrosionsschutzbeschichtung auf einem Substrat ausgebildet wird, dadurch

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21. Procedure according to requirement 20, by the fact characterized that the layer is formed by glowing of the substrate, which consists of chromhaltigem rustproof steel in oxygen-containing atmosphere. 22.Verfahren according to requirement 21, by the fact characterized that the glowing takes place with 800 DEG C to 1,000 DEG C. 23. Anti-corrosive coating for applications in more carburizing (more reducing) atmosphere at high temperatures, whereby the anti-corrosive coating on a substrate is trained, by the fact characterized that on the substrate (1) a thin layer of a not diffusing material is intended as diffusion barrier layer (2) and on the diffusion barrier layer (2) a thin layer of a material corrosion resistant as corrosion protection layer (3). 24. Anti-corrosive coating according to requirement 23, by the fact characterized that the corrosion protection layer (3) has a thickness from 0,2 mu m to 10 mu m. 25.Korrosionsschutzbeschichtung according to requirement 23, by the fact characterized that the corrosion protection layer (3) has a thickness from 0,5 mu m to 5 mu m. 26. Anti-corrosive coating according to requirement 23, 24 or 25, by the fact characterized that the diffusion barrier layer (2) has a thickness from 0,2 mu m to 6 mu m. 27. Anti-corrosive coating according to requirement 23, 24 or 25, by the fact characterized that the diffusion barrier layer (2) has a thickness from 0,2 mu m to 3 mu m. 28.Korrosionsschutzbeschichtung for applications in more carburizing (more reducing) atmosphere at high temperatures, whereby the anti-corrosive coating on a substrate is trained, by the fact characterized that on the substrate (1) a only one thin layer from a not rusting material corrosion resistant is intended as corrosion protection and diffusion barrier layer (3) with a thickness from 0,2 to 10 mu m, preferably 0.2 mu m to 5 mu m. 29. Anti-corrosive coating after one of the requirements 23 to 28, by the fact characterized that the substrate (1) consists of a highly alloyed rustproof steel. 30. Anti-corrosive coating after one of the requirements 23 to 27 or 29, by the fact characterized that the corrosion protection layer (3) exists made of nickel. 31. Anti-corrosive coating after one of the requirements 23 to 27 or 29, by the fact characterized that the corrosion protection layer (3) consists of outer ones, cu, Pt, Ru or Pd. 32. Anti-corrosive coating according to requirement 28 or 29, by the fact characterized that the only corrosion protection and diffusion barrier layer (3) consist of TiN or CrN. 33. Anti-corrosive coating according to requirement 28 or 29, by the fact characterized that the only corrosion protection and diffusion barrier layer (3) consist of chromoxid. 34. Anti-corrosive coating after one of the requirements 23 to 27 or 29 to 31, by the fact characterized that the diffusion barrier layer (2) consists of TiN. 35. Anti-corrosive coating after one of the requirements 23 to 27 or 29 to 31, by the fact characterized that the diffusion barrier layer (2) exists of CrN, ZrN, TiNi or TiCN or made of chromoxid. 36.Stromfuehrendes construction unit for a fusion carbonate gas cell, a in particular anode current collector or a bipolar plate, with a substrate (1) from rustproof steel and an anti-corrosive coating, by, the fact planned on the substrate, characterized that on the substrate (1) a thin layer of a not diffusing material is intended as diffusion barrier layer (2) and on the diffusion barrier layer (2) a thin layer of a material corrosion resistant as corrosion protection layer (3). 37. Energized construction unit according to requirement 36, by the fact characterized that the corrosion protection layer (3) has a thickness from 0,2 to 10 mu m. 38. Energized construction unit according to requirement 36, by the fact characterized that the corrosion protection layer (3) has a thickness from 0,5 to 5 mu m. 39.Stromfuehrendes construction unit according to requirement 36, 37 or 38, by the fact characterized that the diffusion barrier layer (2) has a thickness from 0,2 to 6 mu m. 40. Energized construction unit according to requirement 36, 37 or 38, by the fact characterized that the diffusion barrier layer (2) has a thickness of 0,5 to 3 mu m. 41. Energized construction unit for a fusion carbonate gas cell, a in particular anode current collector or a bipolar plate, with a substrate (1) from high-grade steel and an anti-corrosive coating, by, the fact planned on the substrate, characterized that on the substrate (1) a only one thin layer from material corrosion resistant and a not rusting is intended as corrosion protection and diffusion barrier layer (3) with a thickness from 0,2 to 10 mu m, preferably 0.5 to 5 mu m. 42. Energized construction unit after one of the requirements 36 to 40, by the fact characterized that the corrosion protection layer (3) exists made of nickel. 43. Energized construction unit after one of the requirements 36 to 40, by the fact characterized that the corrosion protection layer (3) consists of outer ones, cu, Pt, Ru or Pd. 44. Energized construction unit

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according to requirement 41, thus that the only Korrosionschutz and diffusion barrier layer (3) exist of TiN, CrN or made of chromoxid. 45. Energized construction unit after one of the requirements 36 to 40, 42 or 43, by the fact characterized that the diffusion barrier layer (2) consists of TiN. 46. Energized construction unit after one of the requirements 36 to 40, 42 or 43, by the fact characterized that the diffusion barrier layer (2) exists of CrN, ZrN, TiNi or TiCN or made of chromoxid. --
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21. Verfahren nach Anspruch 20, dadurch gekennzeichnet, dass die Schicht durch Glühen des Substrats, das aus chromhaltigem nichtrostendem Stahl besteht, in sauerstoffhaltiger Atmosphäre gebildet wird.

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